



PATENT

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Dated: January 14, 2005

BY:

Rodney D. DeKouif  
Rodney D. DeKouif

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: David C. Dunand )  
Serial No: 10/680,639 )  
Filed: October 7, 2003 ) Attorney Docket No. 6513-DIV  
For: SUPERCONDUCTING )  
Mg-MgB<sub>2</sub> AND RELATED )  
METAL COMPOSITES )  
AND METHODS OF )  
PREPARATION )

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

RULE 131 DECLARATION OF DAVID C. DUNAND

1. I, David C. Dunand, am inventor of the invention (the "Invention") disclosed and claimed in the above-entitled application (the "Application"). I am a Professor in the Department of Materials Science and Engineering at Northwestern University. I make this declaration in support of the Application and, in particular, to antedate a reference cited against the Application.

2. The Invention claimed in the Application was completed before the effective date of the journal article by Sharoni, et al., entitled "Spatial variations of the superconductor gap structure in MgB<sub>2</sub>/Al composite," *J. Phys. Condens. Matter* 13 (2001) L503-L508 (*i.e.*, the Sharoni reference). More specifically, the

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JAN-13-2005 03:32P FROM:

Invention was conceived and with due diligence reduced to practice prior to the effective date of the Sharoni reference. (The effective date of publication, i.e., June 4, 2001, can be found at <http://www.iop.org/EJ/toc/0953-8984/13/22>.)

3. This Declaration, and prior invention, is supported by copies of pertinent pages from my laboratory research notebook, entries which were contemporaneously witnessed by Graduate Student Naomi Davis. Date redacted copies of the aforementioned notebook pages are provided collectively as Exhibit A and incorporated herein by reference. These documents establish that the Invention was made at least as early as March 26, 2001, which is a date earlier than the effective date of the Sharoni reference.

I hereby declare that: All statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; that those statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code; and that willful false statements may jeopardize the validity of the Application or any patent issuing thereon.

Date January 13, 2004

David C. Dunand  
David C. Dunand

Monday,

I discussed last week with D.N. Seidman the idea of superconducting composites based on the new compound  $\text{HgB}_2$ . I had mentioned to him the following simple idea:

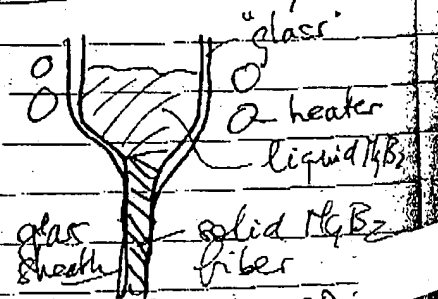
① Infiltrated  $\text{Hg}$ - $\text{HgB}_2$  composites

The  $\text{Hg}$ - $\text{B}$  phase diagram shows that  $\text{Hg}$  and  $\text{HgB}_2$  are at equilibrium with each other. It should thus be possible to infiltrate a packed bed of  $\text{HgB}_2$  powder (which may have been pre-sintered to make a continuous skeleton) with liquid  $\text{Hg}$ . The net result is a composite with a continuous superconducting phase of  $\text{HgB}_2$  embedded in a continuous metallic  $\text{Hg}$  phase, which allows for thermal management (conducting heat away from  $\text{HgB}_2$  and preventing loss of superconductivity) and also being able to carry current if s.c. is lost.

The following other ideas came to me, undisclosed to D.N. Seidman:

② Fabrication of  $\text{HgB}_2$  fibers

Superconducting fibers would be very useful embedded in a non s.c. matrix (polymer, metal, glass, ceramic) which provides thermal and mechanical properties. Existing s.c. fibers can be made by drawing (when ductile) or by powder sintering (when brittle). My idea uses a different technique, i.e. the Taylor wire technique.  $\text{HgB}_2$  powder is packed in a "glass" tube (any amorphous ceramic such as Pyrex, E-glass, etc). The  $\text{HgB}_2$  is melted and the glass is quickly drawn into a hollow fiber containing  $\text{HgB}_2$  which solidifies into a continuous fiber, which can be spooled.



4/20/77 J. Van

Read & understood 1/9/81

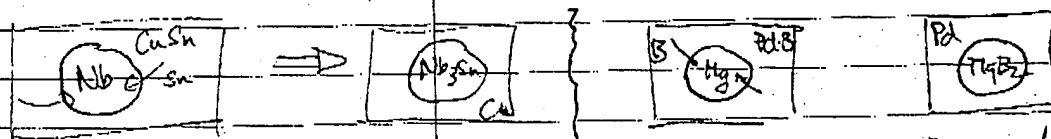
Taylor wires have been made with many metals (see Donald, 1987).

The following requirements are needed: (1) no reaction between glass and  $MgB_2$ , (2) working temperature of glass must be higher than  $MgB_2$ , (3) glass must become highly viscous before  $MgB_2$  solidifies, lest it deforms and breaks the  $MgB_2$ . Pyrex seems a good first choice.

The glass sheathed  $MgB_2$  fiber can then be embedded in a matrix (metal, glass, ceramic, polymer) to form a composite by usual composite processing techniques. Alternatively, the glass can be dissolved by an acid (HF-containing acid) and the bare  $MgB_2$  fibers can be used in a composite. Some dissolution of  $MgB_2$  may be tolerable as part of the glass removal.

### ③ Ductile $Mg-B-Pd$ wires as precursors for $Pd-MgB_2$ composites

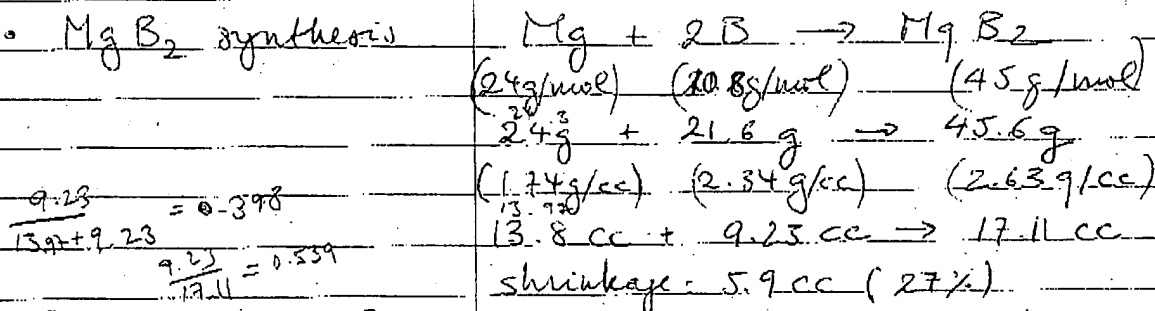
$Nb_3Sn-Cu$  composites are ideal, as Cu prevents thermal runaway. They are produced by sticking Nb wires between a Cu-Sn bronze matrix and drawing, resulting in a ductile Cu-Sn/Nb composite which can be drawn and shaped (coiled, etc.) to shape. Heat-treatment diffuses Sn to the Nb fibers, forming the brittle  $Nb_3Sn$  superconductor in a Cu matrix.



The same idea can be used for  $MgB_2-X$  composites, where  $X$  is a ductile metal which can be alloyed with  $B$ . A good example is  $Pd$ , which can accept 2.2 wt%  $B$  (at%)  $So$ ,  $Mg$  wires could be embedded within sheets of  $Pd-B$  alloy, diffusion-bonded, drawn or shaped as needed. Upon heating below  $650^\circ C$  (melting point of  $Mg$ ),

# calculations for volume fractions of Pd/MgB<sub>2</sub> composite

## • MgB<sub>2</sub> synthesis



$$\frac{9.23}{13.8 + 9.23} = 0.398$$

$$\frac{9.23}{17.11} = 0.539$$

## • Pd-B alloy Pd-2.2wt% B (max solubility)

$$21.6\text{g B} \rightarrow 960.2\text{g Pd}$$

## • Mg-B-Pd alloy

$$960.2\text{g Pd} \quad (12\text{g/cc}) \rightarrow 80.0\text{cc Pd}$$

$$45.6\text{g MgB}_2 \rightarrow 17.11\text{cc MgB}_2$$

83.4 vol% Pd
17.6 vol% MgB <sub>2</sub>

→ 1/6 of the volume is MgB<sub>2</sub>



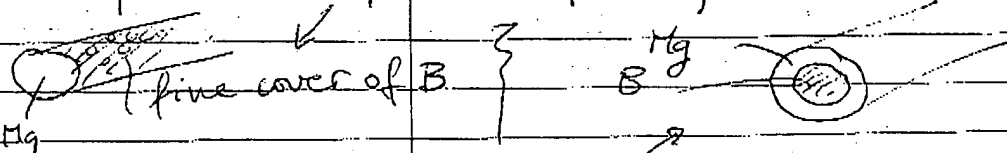
$$\frac{\pi a^2}{4b^2} = 0.176$$

$$\frac{a}{b} = \sqrt{0.176 \cdot \frac{4}{\pi}}$$

This volume fraction is not very high, but still commercially feasible, provided price of Pd is not too high.

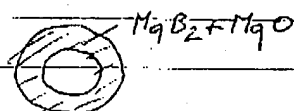
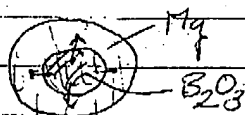
NB: some Mg will dissolve in Pd.

- Additional B could be introduced by dipping Mg wires in B suspension before compacting with Pd or Pd-B foils.

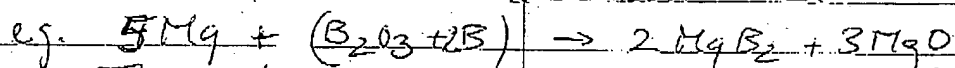


or filling Mg tubes with B powder and then compacting with Pd. Extrusion would still be easy with B powder.  
 read + understood  
 Martin G. Lang

- Alternative is to fill Mg tubes with  $B_2O_3$ , which is viscous at extrusion temperatures. The reaction is then  
 $4Mg + B_2O_3 \rightarrow MgB_2 + 3MgO$



on a suspension of B particles in  $B_2O_3$ , which will remain viscous & drawable.



The advantage is that the metallic matrix can now be a metal different from Pd, and the volume fraction of fibers has no restrictions

$MgO_2$  fibers (160  $\mu m$  in  $\phi$ ) have been disclosed at the APS meeting. B fiber is exposed to Mg vapors and forms  $MgB_2$  which was found to be superconducting.

Taylor wire technique has following advantages:

- much more rapid production
- diameter easily changed (1-100  $\mu m$  typically)
- different grain structure
- sheath of glass.

Good matrix for "filled tube" process: any metal which does not dissolve into Mg (which would contaminate  $MgB_2$ )

1. Cu: zero sol. in Mg; eutectic at 48°C;  $MgCu_2$  &  $Mg_2Cu$ ; 2wt% Mg in Cu
2. Ni: zero sol. in Mg;  $MgNi_2 \rightarrow MgNi$ ; zero

read & understood,

1/20/91

Solubilities

Metals	<del>Sol</del> in Mg	Alloys	Intermetallics	Eutectic (°C)
Cu	0	~3wt%	2	483
Ni	0	0	2	506
Co	0.12	~1.5at%	0	651
Fe	0	0	1	635
Cr	0	0	0	—
Au	0	huge	many	575
Be	0	0	1	—
Pd	0.23	5wt%	7	540
PE	0	0	5	575
Nb	0	0	0	—
Mo	0	0	0	—

Other advantages:

high conductivity: Cu (but reduced by Mg in solid soln)  
 Au (but  $\delta$ )

Processing: Cu low melting  
 electrodeposition: Cu, Ni, Cr, Au, Pd, Pt

no bowdles Cu, Au  
 read & understood  
 Name of line

Other Idea:

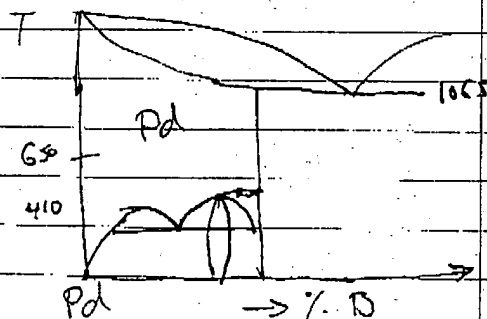
React B with Cu-Mg melt  $2B + Cu + Mg \rightarrow MgB_2 + Cu$   
 this is only possible if Cu does not dissolve and  
 deteriorates  $MgB_2$  s.c. properties

1. infiltrate B fibers or B skeleton  
 with Cu-Mg melt
2. react in the melt
3. solidify Cu-MgB<sub>2</sub> composite.

This will work with non-boride formers, i.e. mostly Cu and Au

Read: 11/11/05

Thinning Jan



pure B dissolves Cu ( $\sim 16 \text{ wt}\%$ ) at 1013°C

1 mol B =  $4.61 \text{ cm}^3 \sim 40 \text{ vol}\%$

$\frac{1}{2}$  mol Mg =  $6.89 \text{ cm}^3 \sim 60 \text{ vol}\%$

30 vol% B }  $\sim 50 \text{ vol}\% MgB_2 \rightarrow \frac{2}{3} MgB_2$   
 45 vol% Mg }  
 25 vol% Cu } 25 vol%  $\frac{1}{3} Cu$



BMG → batch program works! → code  
 some crypt. matrix! → new paper with double  
 no creep error due to reloading after  
 2 hr hold → throw away data  
 stand alone { - compression  
 paper { - hold  
 matrix info

• MRS paper → draft on Monday  
 practice talk on Thursday

Tasker Carr Search Com

↳

↳ carbon steel

Taylor wire MgB<sub>2</sub> trial

(Went to glass blowing shop and tied with pyrex (10 mm)

- can draw easily empty wire

- MgB<sub>2</sub> powder (~ 1 cm high) gets red hot, but no melting visible  
 Can partially draw pyrex, but eventually fractures. MgB<sub>2</sub>  
 does not burn.

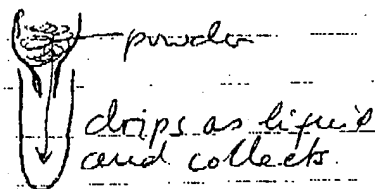
Problem is  $\phi$  is too large, cannot keep the whole block  
 of MgB<sub>2</sub> hot. → try finer tube, maybe try a torch  
 with multiple outlets.

Tried finer wires  $\sim 4 \mu\text{m}$ , and after a few trials, got a wire  $\sim 100-200 \mu\text{m}$ , with black  $\text{MgB}_2$  inside. It is not clear whether the powder was melted throughout, but it seemed to be

• tried Cu in  $4 \mu\text{m}$  tube (pyrex), but not successful due to high  $T_m$  (glass leaked before Cu melted)

• Zn powders worked, nice Taylor wires.

• Tried again  $\text{MgB}_2$  and got a nice length  $\sim 10 \text{ cm}$  uninterrupted. Pyrex was not cleaned, so bubbles interrupt wire in some parts. Maybe also need to "pre-melt" materials, e.g. as done for  $\text{AgCl}$  during their (but  $T_m = 455^\circ\text{C}$  vs  $800^\circ\text{C}$  for  $\text{MgB}_2$ )



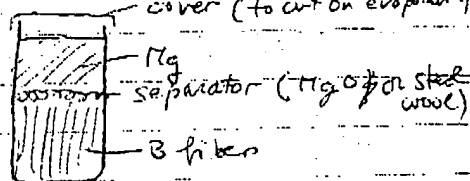
glass shop closed

read Canfield paper (PRL 86, 2423) and had the following

idea: integrated fiber formation & composite fabrication

1. Put B fibers and Mg into crucible

2. Heat at  $950^\circ\text{C} \rightarrow \text{Mg}$  melts and seals crucible  
hold for 2h  $\rightarrow \text{Mg}$  vapors react with B fibers and form  $\text{MgB}_2$  fibers like Canfield's paper



3. Pressurize with gas and force  $\text{Mg}$  into fiber preform  
 $\rightarrow \text{MgB}_2 - \text{Mg}$  Composite

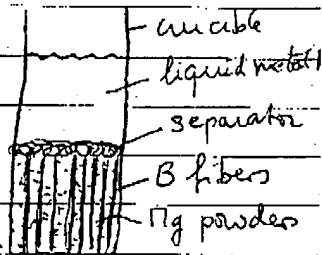
One of the main advantages is that  $\text{MgB}_2$  fibers are formed in situ. They do not have to be handled, bundled, etc, which would easily damage or break them, because Canfield describes them

Another possibility is to infiltrate B-fibers with molten Mg and keep the composite at 950°C for 1-2 h, long enough for complete reaction to form  $MgB_2$ . Solidifying gives a Mg composite with  $MgB_2$  fibers.

#### Method to produce $MgB_2$ metal matrix composites

Campfield's paper shows that  $MgB_2$  fibers can be produced by exposing B fibers to Mg vapors at 950°C for 2 h. However, the fibers are very brittle and bent after fabrication. It will be difficult to bundle them and subsequently infiltrate them without breakage. A solution is to synthesize and infiltrate the fibers in two closely consecutive steps, without handling the fibers.

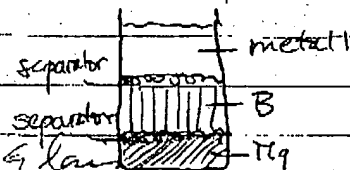
Step 1: Heat-up assembly to synthesis temperature. Mg melts and vaporizes, reacting with B-fibers to form  $MgB_2$ . If the metal 1 is melted, it forms a liquid seal, thus preventing escape of Mg vapors. A non-wetting separator prevents metal 1 from contacting fibers. Examples for metal 1: Mg, Al, Cu alloy with low ~~melting~~ solidus (bronze, brass).



Step 2: pressurize with gas the crucible, forcing liquid metal 1 through the separator and between the  $MgB_2$  fibers, thus making a composite. Solidify and extract composite from crucible. If metal 1 has higher melting point than synthesis temperature of fibers, first raise temperature to melt it.

Complex shapes could be made by this process.

A variation is to put Mg below fibers with separator permeable to Mg vapors. react + under vacuum & low temp.



see p. 136: 24 g Mg  
+ 21.6 g B

13.8 cc Mg 60 vol %  
9.23 cc B ~ 40 vol %

so at best: 20 vol % B

30 vol % Mg powder

50 vol % porosity



$$\phi 5 \text{ mm} \quad A = 19.6 \text{ mm}^2 \rightarrow 3.9 \text{ mm}^2 \text{ B}$$

each fiber is  $100 \mu\text{m} \phi \rightarrow A = \pi \cdot 0.05^2 = \underbrace{0.2 \left( \frac{5}{0.1} \right)^2}_{500 \text{ fibers}}$

drop into 0.3" crucible -

@ 2 cm  $\rightarrow 10 \text{ m}$

Observe Dorian run infiltration

1. Turn H<sub>2</sub>O on (circulate first at ~125 psi)  
open to machine → flow switch on  
zero pressure transducer
2. Close vessel - slide in  
- put PB sheet  
- tighten nut until lid is flush with vessel
3. Connect 1 p-transducer (showing as psix5, full vac = -65.70)
4. TC (check them)
4. Open gas tank, set 2nd stage regulator at 150 psi
5. purge 3 times - use valve 1 only
6. check for vac. leak - install vessel  
verify vac. is constant for 4.5 min
7. Switch on main power, turn to 50% on both variacs  
→ upper zone: 5.5 A / 100 V  
→ lower zone: 4 A / 100 V  
wait ~1½ h → 350°C
8. put T recorder on turn to 75% (top only)  
→ upper zone: 8.5 A / 150 V  
wait ~ 4.5 min → turn to 90% (top only) (70% bottom)  
wait ~ 40 min → turn down to 50% (both)  
and fine-tune  
4.5 min
9. Infiltrate 1. increase to 550 psi (regulator)  
2. open valve #2 to vessel  
3. switch off power  
4. open valve #1 and pressurize to 550 psi (35 atm)  
5. close valves 1 & 2 (will leak a 1-2 psi/min)
10. Cool down ~ 1 h to 200°C  
turn off water, vent (use vent valve)  
open

17g 99.99% 2 pieces from Emma (26.33g)

started cutting pieces

cut tubing & cleaned tubing

tubes: 0.555" ID = 14.10mm  
0.305" ID = 7.75mm

filling in small tube to 2cm:  $V = 943 \text{ mm}^3 = 0.943 \text{ cm}^3 \rightarrow$   
assuming 50% packing:  $0.472 \text{ cm}^3$

$0.472 \text{ cm}^3 \times 2.63 \text{ g/cc} \rightarrow 1.24 \text{ g MgB}_2$   
1.70  $\rightarrow 0.802 \text{ g Mg}$  double  $(1.60 \text{ g Mg})$

for large tube, multiply by  $\left(\frac{0.555}{0.305}\right)^2 = 3.3 \rightarrow 4.1 \text{ g MgB}_2$   
5.3g Mg

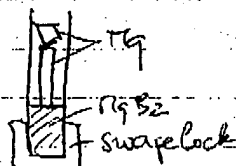
called by ( )  
else will send ~ 30 feet of 4 mil B fibers for free  
Goodfellow charges a \$100 for 10m. [minimum order: 1 lb \$792 for 4 mil  
5mm W core, 100µm diameter, 20.2 mg/m] \$1159 for 6 mil  
 $\rightarrow 10 \text{ m} = 0.202 \text{ g}$

prepare small crucible: 17g:  $(1.605 \text{ g})$  (2 pieces)

add  $\text{MgB}_2$  to crucible bottom far: 47.210g

-62g tap + push with rod  
48.208  
383  
48.441g

add pieces of Mg on top



$\rightarrow 1.231 \text{ g MgB}_2$

measured height: 6.5mm??  
not right, was not "touching  
bottom" at first!

4.5mm for 0.233g  $\rightarrow$  ~ 22.5mm for full mass, OK!

leave for evening

146

Sue Abkowitz

RT E (ksi)	600°F G (ksi)	modules measurement
Ti64 : 16.88	<del>16.88</del>	
Ti64+10W : 15.67 ↳ (5.60g)	5.041 → 5.052 → 5.056 → 5.060 → 5.067	0 5 min 10 15 20 min

-10%

0.02

change 0.4% in 20 min

• No dissolution : no, T too low, G increases

• W burn of : no, " "

(check if Ar is used)

•  $\beta \rightarrow \alpha$  transf. is most likely!mass : 4.633  
volume  $\frac{14.1}{1.5} = 2.35 \text{ cm}^3$ ↳ 1.98 g/cm<sup>3</sup>

Alfa Aesar

prepare large crucible 4.633 g MgB<sub>2</sub> (15 mm height)

5.768 g. Mg - 29.99% (2 large pieces, 1 small)

- put a thin 200 felt layer on top of Mg, stuff crucible to the top w. 200 felt (to prevent powder blow out during vac)
- load sample in vessel, contained in 503 crucible/pot
- evacuate/purge 3 times

11:35 vacuum -76, insulate vessel (leak check)

12:15 up low -75, 20 K, no leaks

12:18 50% 50% T<sub>1</sub> T<sub>2</sub> T<sub>3</sub> T<sub>4</sub> Vac 20.7 -76

12:30 60% 60% 191 -73

1:40 " " 516 -70

2:00 75% 75% 582 568 -70

2:35 65% 65% 742 743 743 (734) -69

2:43 133 137 139 735

2:49 68% 68% 732

2:55 " " 734 737 739 734

switch off power

2 minute resuscitation to 2400 (980 psi)

3:40 depressurize 170°C, 1400 (200 psi)

Mg is at level of TC4

2 crucibles by steel crucible





new large crucible: old  $Hg$  powder from previous exp. 3.475g  
new 0.939

old  $Hg$ : 4.558g 2 pieces  
new  $Hg$ : 2.4704 1 piece

4.417g  
(Alfa Aesar)

7.028g (99.99%)

add  $0.30 \pm 0.05$   $Hg$  to small crucible

both crucibles leave  $Hg$  to the rim, put no felt on top

• load crucible in infiltrator

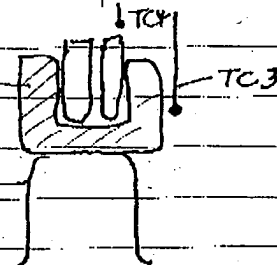
put them in thick graphite crucible  
on top of s.s. crucible

much less insulation as last time  
should get better heating

TC3 is next to gr. crucible

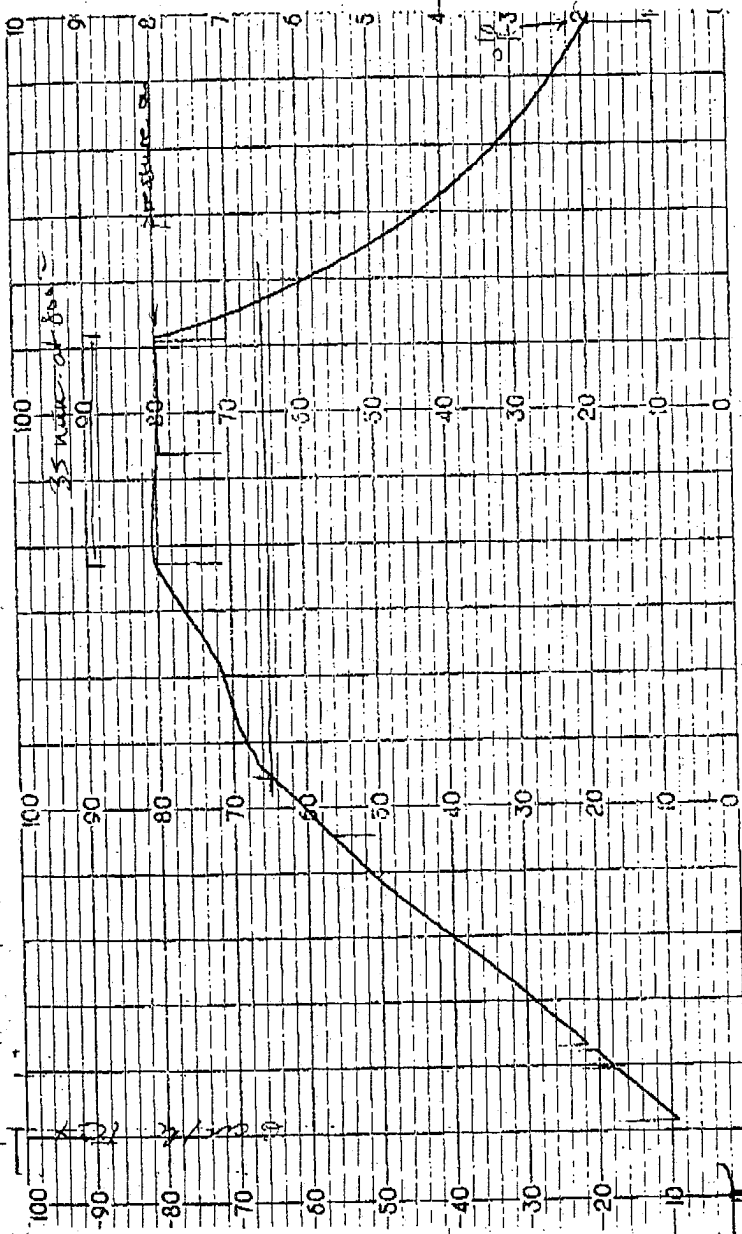
TC4 is just above steel crucibles

• evacuate to -67, insulate & leave for the night 11:35 PM



9<sup>30</sup> AM, vac at -41, good cal. 1 Evacuate & flush twice

	P	T <sub>1</sub>	T <sub>2</sub>	T <sub>3</sub>	T <sub>4</sub>	upper lower	lower higher	
9:55	-55	24	22	19	21	70%	70%	change Ar bottle
10:25	-55	406	382	370	295	"	80%	
10:47	-53	568	569	590	538	75%	85%	
11:14	-51	688	702	732	712	80	95%	
11:30	-50	797	804	825	801	80%	95%	
11:45	-49	795	795	801	799	"	80%	
12:05	-49				803	"	"	switch off, infiltrate
12:07	2525				755	0	0	in GORE
12:10	2232				662			
12:39	1468				280			
12:52	1312				195			pump out, open



Carbon crucible contains 4.0 g of ~~the~~ coarse, grey drose  
could be Mg evaporated and condensed?

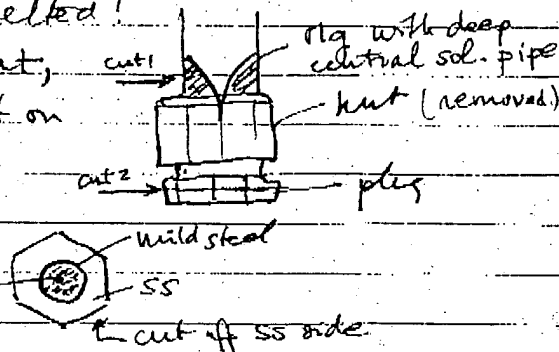
▲ cut large crucible → Mg has melted!

small oxide "hull" left in upper part,  
(very light) <sup>0.43g</sup> Some white deposit on  
top (T<sub>90</sub>?). Cut

Cut 1: hack saw, see pipe hole

Cut 2: diamond saw.

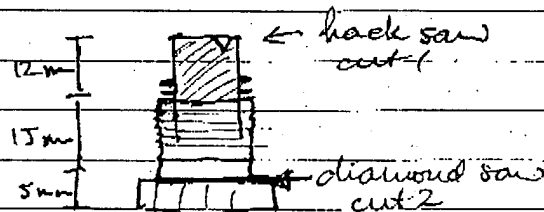
looks infiltrated!



checked under microscope: diamond cut is good enough  
to show that the  $MgB_2$  powders are infiltrated  
with Mg, forming a  $Mg/MgB_2$  composite.  
Took a polaroid, see next page.

▲ open small crucible

• cut 1: Mg to the rim, except  
small blowhole



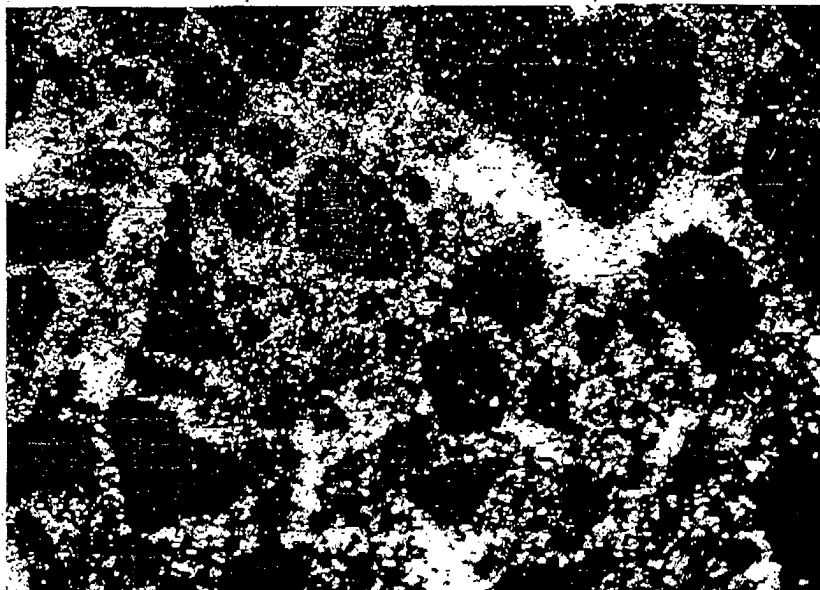
• cut #2 shows only powder, no infiltration

possible problem: 1) leak at cut

2) bad seal by liquid Mg (unlikely)

3) premature solidification (high surface area)

discard sample.



Mg: white  
MgB<sub>2</sub>: black

$\sim 1/3$  Mg  
 $2/3$  MgB<sub>2</sub>  
should have  
continuous  
current  
path!

Polaroid: plug side of cut 2, very bottom of sample  
sample Mg/MgB<sub>2</sub> (1) 40x objective (400x magnification)  
- MgB<sub>2</sub> is agglomerated (size  $\sim 50-100 \mu\text{m}$ )  
- Mg infiltrates as vein between agglomerates (veins  $\sim 10-30 \mu\text{m}$ )  
- many agglomerates are also filled with Mg ( $\sim 1-5 \mu\text{m}$ )  
- some show holes (pull out due to cutting?)

→ this is a fully infiltrated composite, with a few possible regions of porosity (maybe due to closed porosity in original powders or interfering)

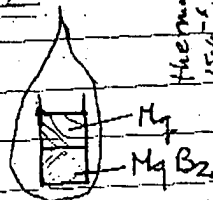
To do: mount & polish this sample

machine steel off

measure conductivity vs T.

next experiments / possible directions)

thermal exp.  
15/16"  $\pm$  1.5%  
OD: 15.895 mm  
ID: 0.24 mm



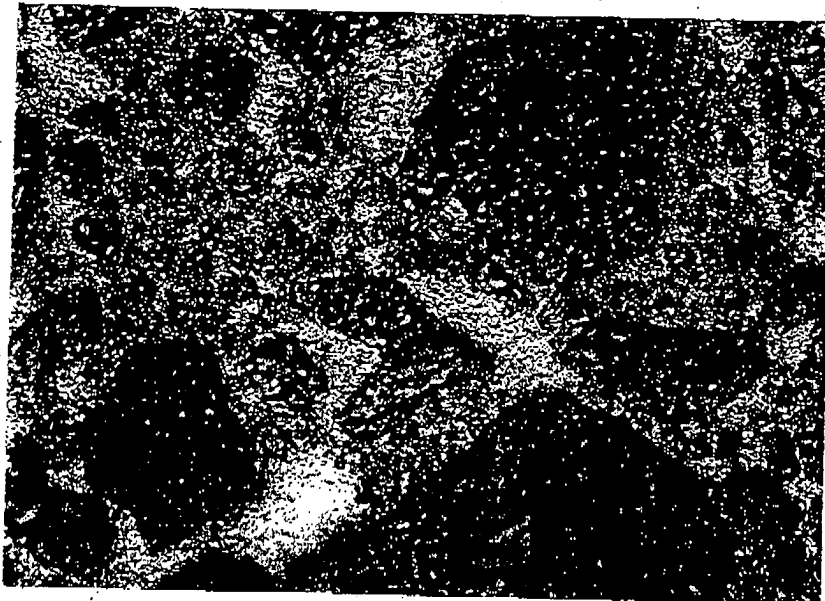
- does  $Hg$  wet  $MgB_2$ ?  
use steel crucible to encapsulate  
 $MgB_2 + Hg$   
also may show enhanced sintering due to  $Hg$  vapors  
(check density of green compact with the pycnometer)
- react B fibers in capsule, then infiltrate
- " B powders " " "
- infiltrate B fibers and then react in molten state
- " B powders " " "
- infiltrate  $MgB_2$  with Al } new crucibles  
" " " Cu } or BN coated steel

polishing same metallographic section (composite MMC)

1. SiC paper with  $H_2O$
  2. 1  $\mu m$  diamond in oil (0.05  $Al_2O_3$  gum up matrix)
- took 2 pictures (see next page)
- composite is successful: 1) full infiltration, no porosity
  - 2) no reaction between  $Hg$  &  $MgB_2$
  - 3) no reaction of either phase w. crucible

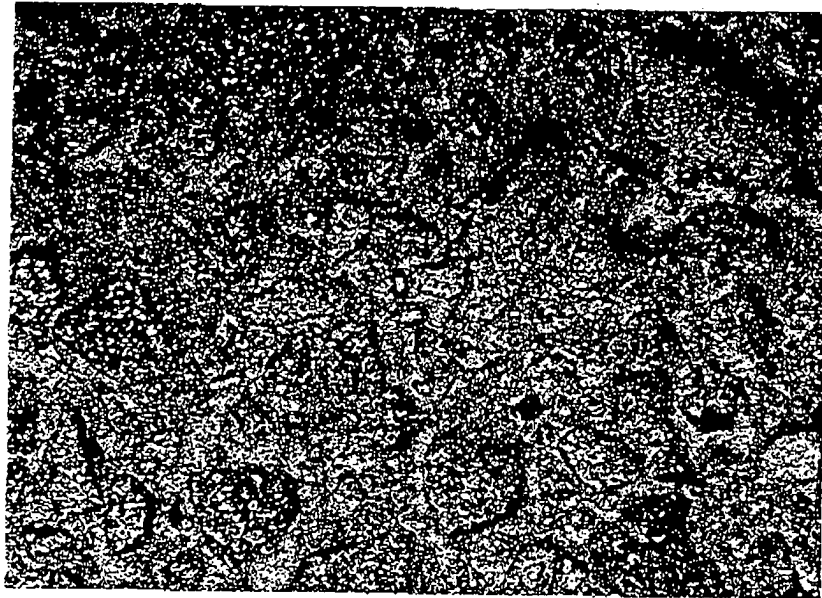
Machine shop: asked for EDM of MMC  
welding of crucibles

Ordered: B powders,  $Hg$  powders, more steel



400x

50 μm



200x

100 μm

at the edge of composite, saw  
pure Mg annulus with some  
interdendritic shrinkage porosity.  
Not seen in composite

- large (~ 20-100 μm) Mg B<sub>2</sub>  
- small (~ 2-20 μm) Mg B<sub>2</sub>



→ need to sieve

can be added in Mg  
matrix

153

Metals which can be infiltrated in current machine

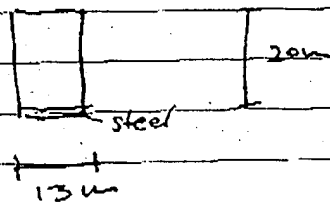
	$T_m$	$T_{ACS}$	
Cu	1088 (?)	100%	too hot!!
→ Ag	962	108%	
Al	662	65%	
<u>Mg</u>	<u>650</u>	<u>39%</u>	
Zn	420	28%	
Sn	232	16%	
(Brass 260 955H)		28%	
" 464 900		26%	
ln	156	20%	
Pb	327	8%	
Cd	321	25%	
Au	1064	73%	too hot!!

Mutual solubilities in Mg-X systems

	in Mg	in X	intermetallics	eutectic
Mg-Mo	0	0	—	—
Mg-Nb	0	0	—	—
Mg-Co	0	0	MgCo <sub>2</sub>	635
Mg-Cr	0	0	—	no phase diag
Mg-Fe	0	0	—	—
Mg-Be	0	0	1	—
Mg-Nd	0	~2% (sat)	4	528
Mg-Pd	0.23%	2.5at%	7	570
Mg-Pr	0	0	5	575
Mg-Ti	0.12%	1.5at%	—	651
Mg-U	0	6 at%	2	483
Mg-Au	0	huge	many	575

mass: 5.213 g

Received machined sample from machine shop. Machinist found composite to be much harder than stainless steel: bridge screws well bonded to matrix.



Side of cylinders show a few pure Hg regions (shiny) but mostly composite

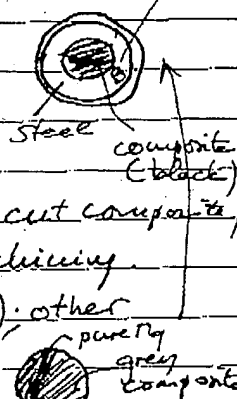
top part was faced → composite (grey)

bottom part is acut from diamond saw

→ need to cut again with diamond saw

The difference of color (grey for machined composite, black for cut composite) is probably due to smearing of Hg during lathe machining.

cut bottom slice (1.5 mm) with diamond saw (or 2h!); other side looks nicely infiltrated, except for a large vein of pure Hg. Unlike the other side, it is not black (could be due to corrosion in). Keep slice and mark it with a "B".  
Actually, due to methanol 1 mm in it produces black color it is probably etched by it!



cut upper slice ( )

final dimensions:  $h: 17.044 \text{ mm}$   
 $D: 12.741 \text{ mm}$   $r = 6.371$  }  $2.1753 \text{ cm}^3$   
 $m: 4.374 \text{ g}$

$\rho = 2.011 \text{ g/cc}$

$$f \cdot 2.63 + (1-f) \cdot 1.74 = 2.011$$

$$f(2.63 - 1.74) = 2.011 - 1.74 = 0.271$$

$$30.5 \text{ vol} \% \text{ Hg B}_2$$



other sides with

large regions of pure Hg, possibly displaced by falling chunks of Hg upon loading in crucible. In this region, volume factor is larger.



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